DEPARTMENT OF THE NAVY
OFFICE OF THE GENERAL COUNSEL

COUNSEL FOR THE
NAVAL RESEARCH LABORATORY
WASHINGTON, D.C. 20375-5000

5870
1208.2

Navy Case No. 70,840

Commissioner of Patents and Trademarks
Washington, DC 20231

Applicant(s): Keith A. Snail et al.

For: INFRARED INTEGRATING SPHERE

Sir:

Transmitted herewith are the papers above-identified constituting a Patent Application filed by the Department of the Navy on behalf of the above-names Applicant(s).

The total filing fee has been computed in accordance with the folloiwng formula:

Basic Fee ----------------------------------$340.00

Additional Fees:

1. Total number of claims in excess of 20 times $12.00 -----------------$_______
2. Number of independent claims minus 3 times $34.00 ---------------$_______

Total Filing Fee ---------------------------$ 340.00

Kindly charge the aforementioned total filing fees and any additional fees due, or credit overpayment of fees, to Deposit Account No. 04-0814.

Respectfully,

THOMAS E. McDONNELL
Reg. No. 26950

Encl:
(1) Application Papers
(2) Information Disclosure Citation

Navy Case No. 70,840

APPLICATION FOR LETTERS PATENT

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT Keith A. Snail and Kevin F. Carr

citizens of the United States of America,

and residents of Washington, DC and Sunapee, New Hampshire

have invented certain new and useful improvements in INFRARED INTEGRATING SPHERE

of which the following is a specification:

COPY

Prepared by:
Edward F. Miles
Tele: 767-3428

Navy Case No. 70,840

INFRARED INTEGRATING SPHERE

Background of the Invention

The optical characteristics of a material is an important material property, and can be used, for instance, to assign optical signatures to well-known objects or classes of objects, and to identify such objects or classes of objects remotely. For an opaque object, i.e., one having zero transmittance, the object's directional emittance can be characterized if one knows the object's directional hemispherical reflectance as a function of object temperatue and angle of incidence. Many systems for determining reflectance are known, prominant among which are integrating spheres, which for decades have been used to measure the reflectance of diffusely reflecting materials in the UV, visible, and near IR. Unfortunately, their exist no generally agreed upon reflectance standards beyond 2.5 micrometers in the infrared. Consequently the reflective properties of materials in the infrared are not well known, and there is a need for integrating sphere systems which can measure the infrared diffuse reflectivity of materials with efficiency, convenience, and reliability. Infrared measurements are complicated by air having several

constituents (e.g. water and carbon dioxide), that absorb at infrared frequencies, which can distort or otherwise make less precise such measurements of diffuse reflectance if the measurements are made in an air atmosphere with a single beam spectrophotometer. Unfortunately, were one to contain any of the present integrating sphere systems in a chamber containing an artificial, non-absortive, atmosphere, one could examine the angular dependence an object's diffuse reflectance only by venting the atmosphere after each test at each angle of incidence, repositioning the object to change the angle of incidence, and recharging the system's artificial atmosphere. This repeated venting and recharging is most inefficient, inconvenient, and uneconomical.

Summary of the Invention

Accordingly, an object of this invention is to provide a novel integrating sphere testing system that can measure the diffuse reflectance of samples in the infrared.

Another object of the invention is to operate the system in an atmosphere that has virtually no absorptance in the infrared.

Another object of the invention is to enable one to reposition samples within the system by means external to it, so that one can make a plurality of measurements to

test the angular dependence of a sample's diffuse reflectance without needing to vent and replenish the system's atmosphere between any of the plurality of measurements.

Another object of the invention is to enable one to selectively vary sample temperature, so at to allow testing of the temperature dependence of the sample's infrared diffuse reflectance.

In accordance with these and other objects made apparent hereinafter, the invention provides an integrating sphere disposed in an airtight chamber under an atmosphere that does not absorb infrared frequencies. The sphere has two positions where a sample may be mounted, one at the sphere's center, another on the sphere's wall, each position corresponding to a different mode by which the sphere can measure diffuse reflectance. In one mode, a rod disposed along a radius vector of the sphere acts as a mounting pedestal for a center-mount sample, disposing the sample at the sphere's geometrical center. The rod is rotatively mounted about its elongate axis so that a center-mounted sample can about with the rod is elongate axis. The rod penetrates the sphere and the airtight chamber, terminating in a handle by which the rod can be rotated as above described, enabling one to select the incidence angle of

the beam on the center mounted sample. In this mode one can reposition the pedestal to systematically examine the angular dependence of a specimen's diffuse reflectance. In addition, a reference or standard can be mounted back-to-back with the sample to be measured. By having the positioning handle of the rod external to the test chamber, one can angularly reposition the sample in the sphere externaly and without the need to vent and replenish the atmosphere in the apparatus for each angular measurement. In the wall-mount mode, a sample is placed on the sphere's wall, and one uses the sphere's wall as the reference. Adjacent to the wall-mount position is a heater for varying sample temperature, with which one can test the temperature dependence of the diffuse reflectance of the wall mounted sample. The ability to mount samples in either of two modes enables one to compare the diffuse reflectance of the center mounted sample against an identical wall mounted sample for purposes of calibrating data taken in one mode by that taken in the other.

Brief Description of the Drawings

A more complete appreciation of the invention and many of the attendant advantages thereof is readily obtained as the same becomes better understood by reference to the

following detailed description, when considered in connection with the accompanying drawings, wherein:

Fig. 1 is a top schematic view, partly in section, of a measuring system employing the instant invention.

Fig. 2 is a view in the direction of lines 2-2 of Figure 1.

Fig. 3 is a detail of the portion of Fig. 1 encircled by line 3-3.

Detailed Description of the Preferred Embodiment

Referring now to the drawings, wherein like reference numerals designate identical or corresponding parts throughout the several views, and with particular reference to Fig. 1, an integrating sphere 1 is shown disposed within an airtight chamber 15 under a non-absorptive atmosphere fed in at 17. Nitrogen is preferred because it has vitually no absorptance in the infrared, and as such provides a far better atmosphere for chamber 17 than, e.g., air, which contains much water vapor and carbon dioxide, each of which has characteristic infrared frequencies, and whose absorbtance would degrade infrared measurements taken by sphere 1. Chamber 15 has an appropriate door (not shown), so that one can get to the interior of sphere 1 between tests. Sphere 1 is a metal shell of, for example, nickel, whose inner surface is

Lambertian (diffusely reflective). Such a surface can be generated by plating a highly reflective (preferably 95% or greater reflectivity) material onto a pre-roughened surface. In a preferred embodiment, the plating on the inner surface of sphere 1 is gold, an especially good choice not only because of its high reflectivity, but also because its optical properties are generally stable with time. The desired roughness (coarseness) is generated by grit blasting or other conventional techniques. The coarseness of this roughening must be such that the height of micro-peaks on sphere 1's inner surface and the distance between such peaks, is of the same order of magnitude as, or larger than, the wavelengths of light to be diffused, and, of course, small with respect to the diameter of any light beam to be input into sphere 1. Surfaces of coarseness appropriate for infrared wavelengths are readily produced with known methods. The gold plate can be applied by any known process, such as chemical ("wet") electroplating. Sphere 1 preferably has a plurality of ports (not shown) which can be closed by conventional removable plugs (not shown) which have inner surfaces geometrically conformable with, and optically identical to, the inner surface of sphere 1. Such ports enable one to practice the "removable cap technique" for

measuring the reflectivity of sphere 1's inner surface, this technique well known to those skilled in the art. One skilled in the art may also selectively place the ports so that they may facilite specular measurements using sphere 1 by acting as specular subtraction ports. As illustrated in Fig. 1, sphere 1 contains a pair of sample mounts 3 and 11, a sample at 3 being disposed at the center of sphere 1 on elongated pedestal 5, and a sample at 11 being disposed on sphere 1's wall. (Fig. 1 additionally shows a sample 4 located at center-mount position 3.) Pedestal 5 penetrates chamber 15 in an airtight manner and has a termination 7 disposed outside chamber 15. Termination 7 is preferably a precision vernier, and enables one to rotate pedestal 5 and a sample 4 mounted at 3 externally of chamber 15 in a place perpendicular to the elongate length of pedestal 5. Pedestal 5 and sample mount 3 should have as small an area as possible, and be coated with the same material as coats the inner surface of sphere 1. A wall-mounted sample at 11 is mounted on the inner wall of sphere 1, and has adjacent to it heating element 12 which, in a preferred embodiment, is a simple resistive (joule) heater. Penetrating sphere 1 so as to be exposed to sphere 1's inner surface is a conventional infrared detector 13 which is disposed immediately above and in line

with the elongate axis of pedestal 5. Pedestal 5 removably penetrates sphere 1 and chamber 15 so that one can remove pedestal 5 and operate the system in the wall-mount mode. Of course, in the wall-mount mode the opening in sphere 1 through which pedestal 5 would extend is closed by a plug (not shown) whose inner surface is geometrically conformable and optically identical to that of sphere 1. Likewise, in the center mount mode wall mount 11 is removed and similarly plugged.

Also disposed within the atmosphere of chamber 15 is a radiation source 19, preferably silicon carbide, which is heated in any conventional manner, e.g., by a resistive (joule) heater (not shown). Source 19 is disposed at the focus of parabolic mirror 21, and upon source 19 being heated, the electromagnetic radiation generated by source 19 is columnated by mirror 21 and directed to parabolic mirror 23, which in turn directs the radiation to variable iris 25 disposed at the focus of parabolic mirror 23. Light passing through iris 25 is reflected off of parabolic mirror 27, which directs the light to a conventional (e.g. potassium bromide) beam splitter 33, which directs a portion of the light to integrating sphere 1 via mirrors 35, 37, 39.

Beam splitter 33 is disposed at a 45° angle to both fixed corner cube 29, and corner cube 32 that can move linearly along direction 30 at a 45° angle to beam splitter 33. The subsystem formed by corner cubes 29, 32, and beam splitter 33, is conventional in the art and is used to determine the frequency content of the light input to sphere 1, and the magnitude of signals at these frequencies, so as to measure the total energy input into sphere 1 during any test. As light from mirror 27 impinges upon beam splitter 33, a portion of the light is directed to corner cube 29, and another portion to corner cube 32. A portion of light reflected from corner cubes 29, 32 is recombined and directed onto mirror 35. By measuring in any known manner the respective distances of corner cube 29 and 32 from beam splitter 33, one knows the phase angle between the two interference signals in the interferometer. With this knowledge, and the interference patttern, one can use conventional Fourier analysis to determine the spectral distribution and intensity of the light incident upon mirror 35, and hence input into sphere 1. Helium-neon laser 31 can also direct light to beam splitter 33 via mirrors 34 and 36. The light from laser 31 is of a precisely known frequency, and hence serves as an excellent standard by

which to determine the position of moving corner cube 32. Sphere 1 can be linearly translated along the line of sight of port 41, mount 4, and mount 11 at least a distance equal to the distance between center mount 3 and wall mount 11 so that, whether one uses the center or wall mount mode, the sample of either mode "sees" an optically identical beam through port 41.

With particular reference to Figure 2, more detail of the center mount 4 is shown, which has ledges 8a, 8b upon which are mounted sample 4a and known reference 4b respectively. Ledge 4a is adapted to align the face of sample 4a with sphere 1's centerline 9, so that a point of the face of sample 4a is coincident with sphere 1's center, and rotating of pedestal 5 rotates sample 4a's face about centerline 9, and sphere 1's center. Sample 4b is similarly mounted, but, as seen in Figure 2, recessed slightly from centerline 9, so that, when pedestal 5 rotates to place sample 4b in the line of sight of opening 41, sample 4b is slightly nearer the incident light beam than would be 4a. Because sample 4b is a reference, one has no need to measure the angular dependence of its reflectance, and experience teaches that this, combined with the small magnitude of offset from axis 9, introduces no significant error into

system measurement. Mount 4 also has lip 6 directly above samples 4 and obscuring the line of sight between the samples and detector 13 (Figure 1). Lip 6 prevents direct reflection from 4a or 4b into detector 13, ensuring that all light incident upon detector 13 is reflected from the surface of sphere 1.

With particular reference to Fig. 3, the details of a preferred embodiment of wall-mount 11 are shown. Sample 14 is releasably held abutingly adjacent to port 20 of sphere 1, in the line of sight of input port 41 (Fig. 1), by arm 18 of coil spring 18. Preferably sandwitched between sample 1 and arm 18 is heater 12 for controlling the temperature of sample 14. Thermocouple 26 is interlocked (by a conventional means not shown) with power supply 20 for heater 12 so that the power output of supply 20 may be automatically adjusted to control the temperature of sample 14 precisely. This configuration also physically isolates heater 12 from sphere 1 by an insulating dead air space, preventing direct heating of sphere 1 which could result in black-body radiation from sphere 1's inner surface.

In operation, one can measure the reflectance of sample material placed either at sphere 1's center 3, or on its wall at 11. When testing a sample disposed in wall mount

11, sphere 1 is initially positioned with the sample of unknown diffuse reflectance mounted at 11. Light from silicon carbide source 19 is directed to port 41, and to the sample, which is positioned at 11 as above described, and the flux reflected by the sample at 11, impinges on the inner surface of sphere 1, the magnitude of which is measured by detector 13 after both single and multiple reflections off the inner surface of sphere 1. Sphere 1 is then pivoted in direction 43 about port 41 so as to remove the sample and mount 11 from port 41's line of slight in favor of a portion of sphere 1's gold inner surface, and the measurement repeated. (Direction 43 is perpendicular to the plane formed by the elongate length of pedestal 5 and the line of sight between port 41 and sample 4--the plane of the drawing sheet on which Figure 1 is set forth.) Pivoting sphere 1 in this manner maintains the symmetry between detector 13 and the sample of the first measurement, and detector 13 and the portion of sphere 1's surface of the second measurement, thus reducing systematic error. In this manner the relative diffuse reflectance of the wall mounted sample and sphere 1's gold surface is measured, and the reflectance of the gold surface is determined using the "removable cap technique" which can now serve as the reference for later measurements.

Thus calibrated, one can repeat this procedure to measure any sample at 11 of unknown diffuse reflectance using the inner surface of sphere 1 itself as the reference. Heater 12 can vary the temperature of a sample at 11 to allow one to investigate the temperature dependence of such a sample's diffuse reflectance.

For a sample center-mounted at 3, one inserts pedestal 5 carrying sample 4 as above described. Manipulation of termination 7 of pedestal 5 sets the angle of incidence at which light passing through opening 41 hits sample 4. In this manner, a series of measurements of sample 4 may commence by turning standard portion 4b toward opening 41 (hence opaqueing portion 4a) and using the standard to calibrate the system. Thereafter, sample portion 4a may be turned towards opening 41, and a series of measurements taken at various angles of incidence so as to test the angular dependence of the sample portion 4a's diffuse reflectance. Because incidence angle is set by external termination 7, one need not purge and replenish the nitrogen atmosphere in chamber 15 between each re-setting of angular position, a great economy as well as a great convenience to the operator.

SPIE Conference on Infrared Optics, vol. 643, [illegible] 1986 (Orlando, FL)

Optical design of an integrating sphere-Fourier transform spectrophotometer (FTS) emissometer

Keith A. Snail
Optical Sciences Division, Code 6520, Naval Research Laboratory
Washington, DC 20375-5000

Kevin F. Carr

Labsphere, Inc.
P.O. Box 70
North Sutton, New Hampshire 03260

Abstract

This paper describes the optical design of an integrating sphere - Fourier Transform Spectrophotometer (FTS) instrument for measuring diffuse IR reflectance as a function of angle, temperature and wavelength. The integrating sphere is 5 inches in diameter with a center mounted sample stage permitting beam incidence angles of 10 to 70 degrees. Samples can be mounted back-to-back for relative measurements and ports are included for specular subtraction of the reflected beam at 20 and 60 degree incidence angles. A heater capable of producing temperatures over 150°C has been included in a sample mount on the wall of the sphere. In addition, the sphere can be rotated about the beam port, permitting operation in both the center and wall mounted modes. Two detectors are planned for the sphere: a 16 mm^2 square cooled HgCdTe detector and an uncooled 3 mm diameter DTGS detector which is coupled to the sphere with a nonimaging compound elliptic concentrator (CEC). The CEC restricts the detector's field-of-view (FOV) to uniform contrast areas on the sphere wall with essentially no change in detector flux. The sphere's coating consists of a 0.5 micron thick gold film on a aluminum substrate, with a mean feature size of approximately 20 µm; a similar coating with a roughness average of approximately 10 µm was also considered. Measurements of the 10 µm coating's total spectral reflectance from 0.3 - 20.0 µm and the bi-directional reflectance distribution function (BRDF) at 3.8, 10.6, and 20.0 µm are presented. The BRDF results show a Lambertian character in fixed azimuthal planes and no specular peaks until the wavelength equals 20 µm and the incident angle is 80 degrees.

Historical perspective

The integrating sphere concept was first suggested by W. E. Sumpner[1] in 1892. He demonstrated that the wall brightness in an integrating sphere is proportional to the total radiation emitted by a source placed inside the sphere. Eight years later, R. Ulbricht[2] proposed a photometer based on an integrating sphere. Since that time, a considerable amount of progress in the development of integrating spheres has occurred, however, it was not until 1955 that the first correct derivation of the throughput of an integrating sphere was performed by Jacques and Kuppenheim[3]. In the 1960's Edwards and his collaborators reported on a number of new reflectometer designs including the first visible/NIR sphere with a center mounted sample stage[4]; this work was the basis of a commercial sphere marketed by Gier-Dunkle[5] for the Beckman DK-IIA visible/NIR dispersive spectrophotometer.

In an effort to extend the use of integrating spheres into the mid infrared region, sulfur coatings were investigated by several researchers[6,7]. The preferred coating for infrared integrating spheres now appears to be roughened gold. In 1976, Willey[8] described a sophisticated dual beam instrument which coupled a Fourier Transform Spectrophotometer (FTS) to a diffuse gold integrating sphere for infrared measurements. More recently, Gindele and his collaborators[9] have reported on a diffuse gold sphere - FTS instrument with a spectral range of 2.5-15 microns. In this paper we report on the first mid to long wave (2.5-20 microns) infrared integrating sphere with a center mounted sample stage. In addition, we show how one can estimate the error introduced by a detector viewing nonuniform illuminated areas of the sphere wall. Finally, design curves are developed for coupling detectors to integrating spheres with compound elliptic concentrators (CEC's) and a technique for modifying the CEC by refraction in a cover window is demonstrated.

Design details

In this section we describe the design of a self-contained sphere accessory for the Mattson[10] Cygnus-25 FTS. The NRL Cygnus-25 (see Fig. 1) is equipped with a water cooled 1300°C SiC source, a computer controlled iris, a Michelson interferometer with one arc second (4.85 microradians) corner cubes, a KBr beam splitter, and a 2 x 2 mm pyroelectric detector fabricated from a single crystal of deuterated triglycine sulfate (DTGS). The

10/9/87

major sub-assemblies of the sphere accessory are the sphere itself, the detector and it associated optics, the diffuse gold coating, and the transfer optics.

Sphere geometry

The sphere diameter is 5 inches with six circular ports provided for center or wall mounted samples, a detector, the beam, and two specular subtraction angles. Total open port area in the center mounted mode is normally 1.42 in^2, or about 1.8% of the sphere's surface area. Table 1 details the critical dimensions of the sphere and its ports.

Table 1. NRL integrating sphere characteristics

Sphere diameter (inches)	5.00
Entrance port diameter	1.25
Exit port diameter	0.50
Specular subtraction port(s)	1.25
Wall mounted sample port	0.875
Coating reflectance (est.)	0.95

Specular subtraction with a light trap is possible at 20° and 60° for center mounted samples and 10° for wall mounted samples. The wall mounted sample port also has a ceramic heater and digital temperature controller capable of achieving 150°C sample temperatures. A variable angle mount positions samples at the geometric center of the sphere; the sample holder accepts one inch diameter samples up to 0.062" thick. The mount inserts horizontally into the sphere in order to accomodate a horizontal looking detector at the detector port, which is diametrically opposite the center mounted sample port. The gold coated sample holder also accepts a diffuse gold reference plate, mounted back-to-back with a test sample. Samples are held in a slightly recessed position so that the sample is not within the detector's field-of-view (FOV). A rotary feedthrough allows sample rotation through 360° without breaking purge. For wall mounted operation, the sphere is pivoted by 10° about the beam port, giving a 10° incidence angle on the sample. In the unpivoted position, the beam strikes the sphere wall above the sample.

Detector and associated optics

The standard detector for the Cygnus-25 has a 4.0 mm^2 square DTGS element with a D* of greater than 2×10^8 $cmHz^{1/2}W^{-1}$ at 2 kHz. Currently a 16 mm^2 square Mercury-Cadmium-Telluride (MCT) detector and a circular DTGS/nonimaging cone system are being fabricated. The MCT detector has a peak D* of $5.17 \times 10^9 cmHz^{1/2}W^{-1}$ at 1.0 kHz.

When using integrating spheres, it is important to shield the detector from any high contrast areas on the sphere wall. Without this restriction on the detector's FOV, two samples with identical total reflectances but different BRDF's could give significantly different results[11]. For an integrating sphere with a center mounted sample, a specular reflection from a sample will trace out a ring on the sphere wall as the sample rotates. The detector could be shielded from this high contrast ring with a baffle inside the sphere or a collimator outside the sphere. Visible integrating spheres occasionally use diffuser plates (typically opal glass) over the detector port for this purpose, however in the infrared diffusers are not commonly available. Baffles were viewed as undesirable because of possible distortions of the radiation field inside the sphere.

A nonimaging cone has been designed which permits a restricted field-of-view on the detector with essentially no change in flux. The proper reflector shape to use is a Compound Elliptic Concentrator[12] (CEC), with corrections for refraction through the detector's cover window. Recently Tardy[13] has calculated the throughput advantage of a CEC cone without a cover window versus an optimized collimator. In the next section we extend this calculation to include refraction effects in a cover window and we present a set of general design curves for a windowless CEC's length and entrance aperture as a function of the CEC's field of view and the detector size.

Coating properties

Two roughened gold coatings were considered, both consisting of a 0.5 micron thick gold film on a roughened aluminum substrate. The roughness average of the first surface is approximately 400 micro inches (10 µm) and the second surface appears to have mean feature sizes about 2.5 times larger. Scanning electron micrographs of the two surfaces are shown in Fig. 2. Measurements of the 10 µm coating's total spectral reflectance form 0.3 - 20.0 µm were performed at Surface Optics Corporation[14] using an integrating sphere in the visible /NIR spectral region and a hemi-ellipsoid reflectometer operated in the reciprocal mode from 2.0 -20.0 µm. The results of these measurements are shown in Fig. 3. Note the dip in the reflectance beyond 2.0 µm; this is the wavelength at which the integrating sphere is replaced with the hemi-ellipsoid. It is unlikely that the surface roughness or wavelength

dependence of the gold reflectance is causing this 5-7% drop in the reflectance. A more plausible explanation appears to be non-uniformities in the radiation source of the hemi-ellipsoid; a solution to this problem is proposed in an accompanying paper[15]. A set of comparative measurements at Labsphere on the visible/NIR reflectance of the two coatings indicated that the rougher coating had a higher total reflectance, and on the basis of these measurements the rougher coating was selected for the NRL sphere.

The bi-directional reflectance distribution function (BRDF) of the 10 μm coating was measured for 20, 50 and 80° incidence angles at 3.8, 10.6, and 20.0 microns. For the most part, these measurements show a Lambertian character to the scattering, with a specular peak first appearing at 20 μm and large incidence angles (>50°). The surface also shows a tendency to forward scatter, as illustrated in Fig. 4. Note the lack of a specular peak at all azimuthal angles. Preliminary BRDF data on the rougher coating do not appear to be as Lambertian as the data in Fig. 4.

Transfer optics

Normally, the Cygnus comes with a 15 cm focal length (f#/3), diamond turned, off-axis paraboloidal mirror which provides a 90° beam excursion to the image plane, located in the center of a smaple transmittance mount. Beam diameter in this plane is determined by the source diaphragm diameter in the object plane and the conservation of entendue. The source diaphragm diameter is computer controlled in 1% steps up to a mximum diameter of 1.5 cm; system magnification is normally 3.3.

Since a single diamond turned ellipsoid which would satisfy the beam divergence requirement is not readily availble, we have designed a three mirror system (see Fig. 1) having an effective focal length of 29 cm and a magnification of 5.7. This arrangement uses one concave spherical and two flat mirrors, all of which have front surface aluminum coatings. The resultant beam divergence is less than ±5°, as recommended by the Commission Internationale d'Eclaragie (CIE)[16].

Detector optics comparison

When coupling detector to integrating spheres, it is necessary to restrict a detector's field of view (FOV) so as to prevent direct observation of the beam port, the sample, and the first specular reflection (if present) from a sample. Olson and Pontarelli[17] observed 4-7% variations in reflectance when a reflective, grooved sample mounted on the wall of an integrating sphere was rotated about an axis perpendicular to its surface. This is equivalent to holding a sample's total reflectance constant and varying the BRDF. The highest reflectances were observed when the sample grooves reflected the incoming beam onto the portion of the sphere wall viewed by the detector. Obviously, the accuracy of diffuse reflectance measurements made with an integrating sphere will depend on the degree to which the radiation field within the detector's FOV is uniformly Lambertian.

Contrast calculations

The contrast ratio between a directly irradiated area on a sphere wall and the exit aperture irradiance can be written in terms of the incident flux ϕ_i, the sphere throughput τ, the irradiated spot area A_x, and the exit port area A_e, as follows

$$K = (\phi_i/A_x) \,/\, (\tau\phi_i/A_e) + 1 \qquad (1)$$

By considering the successive reflections of radiation entering a sphere and the fraction of radiation lost through ports after each reflection, Goebel[18] has shown that the throughput of an integrating sphere is given by:

$$\tau = f_e p_w / \left[1 - p_w(1 - f_j)\right] \qquad (2)$$

Where f_e is equal to the ratio of the exit port area to the sphere wall area (A_e/A_s) and f_j is the total port area (beam + detector) divided by the sphere wall area $(A_e + A_i)/A_s$. Equation (2) assumes that the sphere wall is illuminated directly, that the wall's reflectance, p_w, is Lambertian and constant over the sphere, that the reflectivity of all ports is zero, and that no sample is present in the sphere. When the wall reflectivity is set equal to unity, Eq. (2) correctly reduces to $A_e/(A_e+A_i)$. If baffles or sample holders are introduced into the sphere then A_s must be increased to take into account the additional surface area. In the absence of multiple reflections the sphere throughput would simply be $f_e p_w$, the additional term in the denominator corresponds to the throughput enhancement due to radiation undergoing at least two reflections. For the NRL sphere described earlier with $p_w = .95$, Eq. (2) gives a throughput of .028. Substituting Eq. (2) into Eq. (1) gives:

$$K = (A_s/A_x)\left[1 - p_w(1-f_j)\right] / p_w + 1 \qquad (3)$$

Thus, for the NRL sphere with $A_x = 2A_e$, Eq. (3) gives a contrast of 18. This type of analysis can be used to estimate the effect of a detector viewing the beam spot directly. Let the fraction of the sphere wall viewed by the detector which is directly irradiated by the beam by f_{xw}. Then the fractional change in flux at the detector port due to moving the beam spot in and out of the detector's field of view is given by:

$$\Delta\Phi_e/\Phi_e = f_{xw}(K - 1) = f_{xw}\frac{A_e}{\tau A_x} \qquad (4)$$

Hence, shifting a beam spot of area $2A_e$ in and out of the field of view of a detector viewing half the NRL sphere wall would alter the sphere throughput by 17%. This is a very large effect which must be considered carefully when designing integrating spheres.

Dectector optics throughput comparison

In this section we compare the throughput of a baffle, a collimator, and a compound elliptic concentrator (CEC) (see Fig. 5). We have been unable to locate exchange factors for cones, however it is expected[12] that a cone's throughput will considerably lower than a CEC but higher than a collimator. Consider a sphere of radius R and a detector of radius r_d which is constrained to view a virtual source of radius r_s through an aperture of radius r_a (see Fig. 5d). The detector is displaced a distance L from the sphere wall. If we assume that the sphere coating is a perfect Lambertian scatterer, then we expect that the brightness, B, over any surface inside the sphere will be uniform, excluding regions directly illuminated by the beam. The flux at the detector port will thus be $\pi B(\pi r^2)$ and the fraction reaching the detector will be

$$\Phi_D = \pi B(\pi r^2)F_{AD} \qquad (5)$$

where F_{AD} is the exchange factor for radiation leaving the detector port and arriving at the detector surface. One could also write Eq. (4) as a product of the flux incident on the sphere, the sphere throughput, and the exchange factor F_{AD}. A useful expression for evaluating exchange factors for coaxial circular disks is

$$F_{AD} = (D_L - D_S)^2 / 4r_A^2 \qquad (6)$$

where D_L and D_S are the lengths of the long and short meridional diagonals from one edge of the detector port to the an edge of the detector. We assume that the inner surfaces of the collimator and baffle are black and that one is free to vary their apertures as long as the length is adjusted appropriately. For the collimator, F_{AD} has an optimal value, which we have calculated as a function of source and detector size, whereas for the baffle we have limited the aperture diameter to that of the detector to minimize shadowing effects.

The CEC is a surface of revolution of two elliptical arcs with foci at opposite edges of the source and detector. For meridional rays originating on the source, the CEC throughput is essentially unity, less reflection losses, whereas for meridional rays originating off the source the throughput drops to zero. Some skew rays from the source are turned back, leading to a slight rounding of the CEC's field of view. If the CEC is designed to expell flux over the hemisphere and if air surrounds the source and detector, then the CEC concentration and the exchange factor F_{AS} are related by:

$$CF_{AS} = 1 \qquad (7)$$

where $C = (r_s/r_d)^2$. Thus the concentration of the CEC exactly compensates for the CEC's restricted field of view, and the detector flux is equivalent to that obtained with a detector mounted on the wall of the sphere. Since the sphere throughput given by Eq. (2) varies approximately linearly with the detector area (for small f_d), one may want to consider using not just a single CEC but an array of CEC's to increase the throughput. In this case the CEC entrance apertures can be merged so as to fill the detector port completely.

The larger incidence angles of radiation striking the CEC's detector compared to a collimator will undoubtably have a lower net throughput due to higher Fresnel reflection losses. If we assume that the radiance on the detector is Lambertian, then we can estimate the size of this effect by calculating an average absorption as a function of the half angle of the radiation cone illuminating a point on the detector. The results of such a calculation are shown in Fig. 6 for detector optical constants typical of MCT at 10μm (n,k = 3.5, .2). Although the reflectivity falls rapidly beyond 60°, the projected area factor desensitizes the average absorption to this decline. The overall effect appears to be less than 3%.

Figure 7 shows a throughput comparison for the CEC, the collimator, the optimized

collimator, and the minimal baffle ($r_{\bullet}=r_d$). The results are normalized by the CEC flux and plotted vs. the source/sphere radii (r_s/R = 1 is a great circle source). The standard Cygnus-25 DTGS detector is assumed, giving r_d/R=.0178. Note that for our design case (r_s/R=.9), a factor of 5.6 improvement in throughput over the optimized collimator is predicted for the CEC. As the solid angle subtended by the source expands towards 2π, the different detector coupling shcemes converge to the same throughput.

Figure 8 shows the length and aperture of a windowless CEC mounted on a sphere versus the source size, plotted on a logarithmic scale. The upper limit on the CEC aperture is equal to the source radius, and as this limit is approached the CEC length goes to infinity. For the range of detector sizes plotted, r_s/R values less than 0.5-0.7 result in CEC lengths comparable to the sphere diameter. Thus, it appears that the CEC may not be suitable for applications requiring very narrow fields of view, such as those encountered with spheres operated in the reciprocal mode.

Refraction compensation

The proper shape for the CEC arc is not actually elliptical, due to refraction in the detector assembly cover window. One can argue that the blurring of the edges of the source due to refraction will be of the order of the thickness of the window or less, and consequently the lliptical shape should be retained for ease of fabrication. For applications where source dimensions need to be precisely controlled, the CEC's shape can be modified with a general string technique[12]. We have performed such a modification and are initiating fabrication of a refraction compensated CEC on a computer numerically controlled (CNC) lathe. The detector will be a circular DTGS detector[20] with approximately 75% more surface area then the standard Cygnus-25 DTGS detector. Figure 9 shows a comparison of a refraction compensated CEC and a wall mounted windowless CEC for identical detector and source sizes. Note that the refraction compensated CEC is approximately 5% longer with a 4% increase in concentration, compared to the windowless CEC.

Signal to noise ratio

In the previous section we discussed the throughput of integrating spheres including the effect of the detector's optics. The overall throughput of an FTS-integrating sphere combination also includes factors relating to the source, interferometer and transfer optics. In this analysis we will assume these factors are fixed and start with the beam flux entering the sphere. The signal from the detector can then be written as:

$$S = \phi_i \, \tau_{sph} F_{ad} \tag{8}$$

where ϕ_i the beam flux entering the sphere. Noise originating in the detector can be written as:

$$N = \sqrt{(A_d \nu)} \, / \, D^* \tag{9}$$

where D* is the average detectivity of the detector material in (cm $Hz^{1/2}/W$) and ν is the scanning frequency, which is normally 4 kHz for the Cygnus-25 DTGS detector. Hence the overall signal-to-noise ratio of the instrument will be:

$$S/N = \phi_i \, \rho_w / \left[1 - \rho_w(1-f_j)\right] f_e F_{ad} D^* / \sqrt{(A_d \nu)} \tag{10}$$

where we have not assumed anything about the nature of the detector optics. The beam power in the sample compartment was measured to be 10 mW with a pyroelectric radiometer. F_{ad} for the 4 mm^2 DTGS detector was calculated to be .026, assuming a window diameter of .410 inches and a detector-to-window distance of .160 inches (the detector port diameter was adjusted downward to the window diameter for the sphere throughput calculation). If we assume a conservative D* of 1 x 10^8cm $Hz^{1/2}/W$, then eq. (10) gives 58 for the signal to noise ratio. By adding a CEC to a circular DTGS detector of equivalent area, this increases to 214, or by a factor of 3.7. This improvement is somewhat less than that predicted earlier, and is due to the wider field of view of the standard DTGS unit compared to that assumed for the CEC (r_s/R =0.9). Even so, the advantage of using a CEC is obvious. A similar calculation for the MCT detector yields a signal to noise ratio of 200-285, depending on the value used for the average D*.

Conclusions

When dealing with integrating spheres having center mounted samples, a compund elliptic concentrator (CEC) is the optimal way to couple a detector to the sphere. The CEC permits restricted fields of view with negligible losses in detector flux. Because the throughput is frequently detector size limited in the infrared, it may be useful to eomploy arrays of DEC's with their detectors connected in series.

BRDF data on the Labsphere 400 μinch roughened gold coating show negligible specular behavior for wavelengths ranging from 2-20 μm. The somewhat low directional reflectance measurements are being checked at another laboratory[11] and will be reported in a future publication. Preliminary data on the newer gold coating indicate that while its reflectivity is higher, the BRDF is not as uniform. SEM photographs show horizontal feature sizes about 2-3 times as large as the 400 μinch coating.

Acknowledgements

At NRL, Keith Snail would like to acknowledge Arthur Morrish for his assistance in the operation and testing of the instrument. At Labsphere, we wish to thank Richard Ellis for his creative implementation of the sphere design and Phil Lape for underwriting part of this research. We also thank Ternay Neu at Surface Optics Corporation for his interest in our project and assistance with the characterization of the roughened gold coating. Finally, we are grateful to Joel Covey at Mattson Instruments for his suggestions regarding the transfer optics, detectors, and other matters. This research was supported in part by a grant from the Defense Advanced Research Projects Agency.

COPY

References

1. W. A. Sumpner, Phys. Soc. Proc. 12, 10 (1892).
2. R. Ulbricht, E. T. Z. 21, 595 (1900).
3. J. A. Jacques and H. F. Kuppenheim, J. Opt. Soc. Am. 45, 460 (1955).
4. D. K. Edwards, et. al., "Integrating sphere for imperfectly diffuse samples", Appl. Opt. 51, 1279 (1961).
5. Gier-Dunkle has now become Dynatech.
6. J. T. Agnew and R. B. McQuistan, J. Opt. Soc. Am. 43, 999 (1953).
7. W. G. Egan and T. Hilgeman, "Integrating spheres for measurements between 0.185 μm and 12 μm", Appl. Opt. 14, 1137 (1975).
8. R. R. Willey, "Fourier transform spectrophotometer for transmittance and diffuse reflectance measurements", Appl. Spectr. 30, 593 (1976).
9. K. Gindele, et. al., "Spectral reflectance measurements using an integrating sphere in the infrared", Appl. Opt. 24, 1757 (1985).
10. Mattson Instrument, Inc., 6333 Odana Road, Madison, WI 53719.
11. Observed at Labsphere with the Lockheed SRMS vacuum integrating sphere spectrophotometer. Contact Rich Osiecki at Lockheed Palo Also Res. Ctr.
12. W. T. Welford and R. Winston, "The optics of nonimaging concentrators: Light and solar energy", Academic, New York (1978), p. 75.
13. H. L. Tardy, "Flux concentrators in integrating sphere experiments: potential for increased detector signal", Appl. Opt. 24, 3914 (1985).
14. Surface Optics Corporation, Hibert St., Suite C, San Diego, CA 92131.
15. K. A. Snail, "New optical systems for the measurement of diffuse reflectance", SPIE Conference on Infrared Optics, April, 1986, Orlando, Fla.
16. Optical Radiation Measurements, vol. 2, edited by F. Grum and C. J. Bartleson, Academic (New York), 1980, p. 70.
17. O. H. Olson and D. A. Pontarelli, "Asymmetry of an Integrating Sphere", Applied Optics 2, 631 (1963).
18. D.G. Goebel, "Generalized integrating sphere theory", Appl. Opt. 6, 125 (1967).
19. Handbook of Heat Transfer, ed. by W.M. Rohsenow and J.P. Hartnett, McGraw-Hill, New York, (1973).
20. Infrared Associates, Inc., 12A Jules Lane, New Brunswick, NJ 08901.
21. Willey Corporation, P.O. Box 670, Melbourne, Fla. 32901

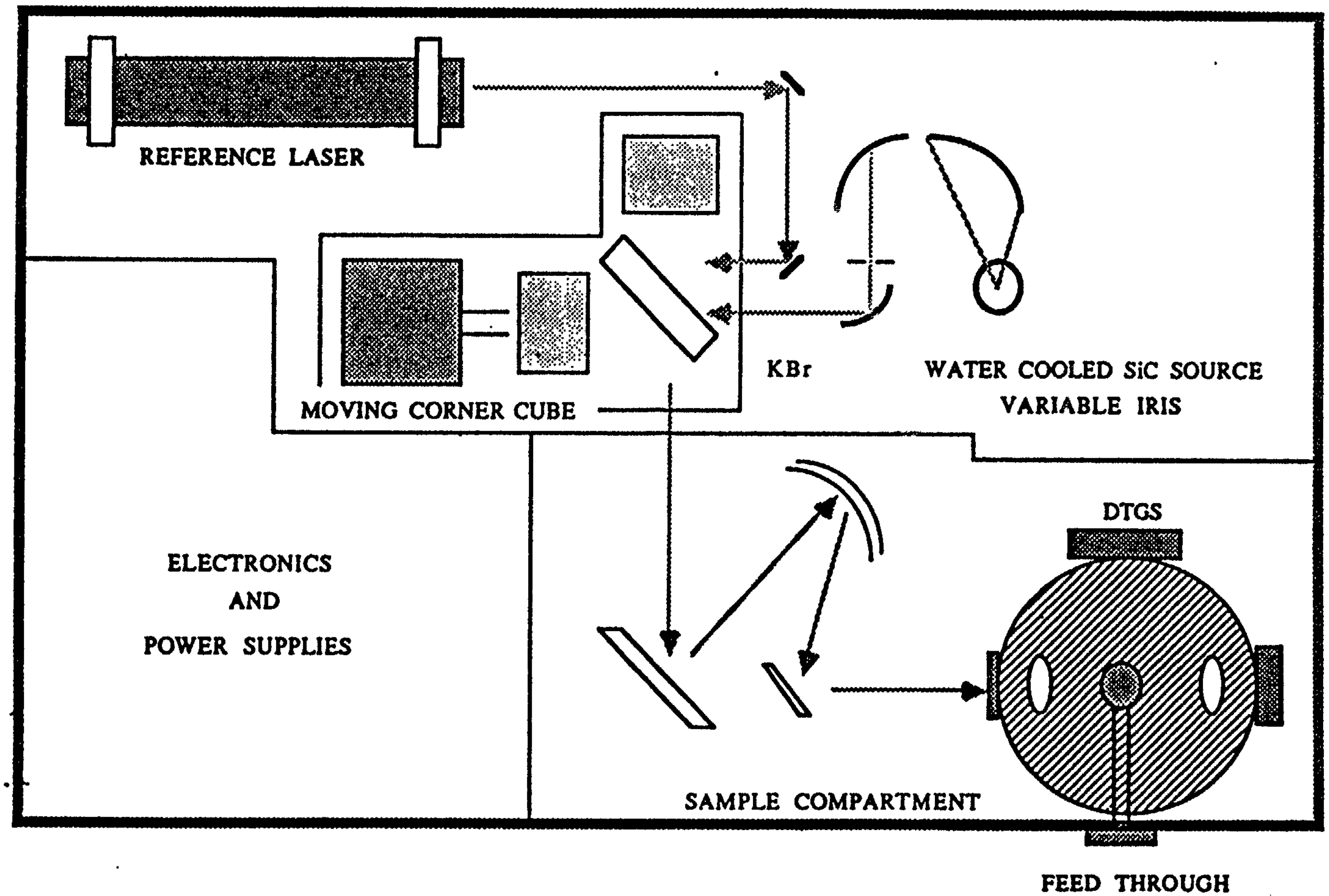

Figure 1: Layout of optical bench in Mattson Cygnus-25 Fourier Transform Spectrophotometer with Labsphere integrating sphere accessory installed.

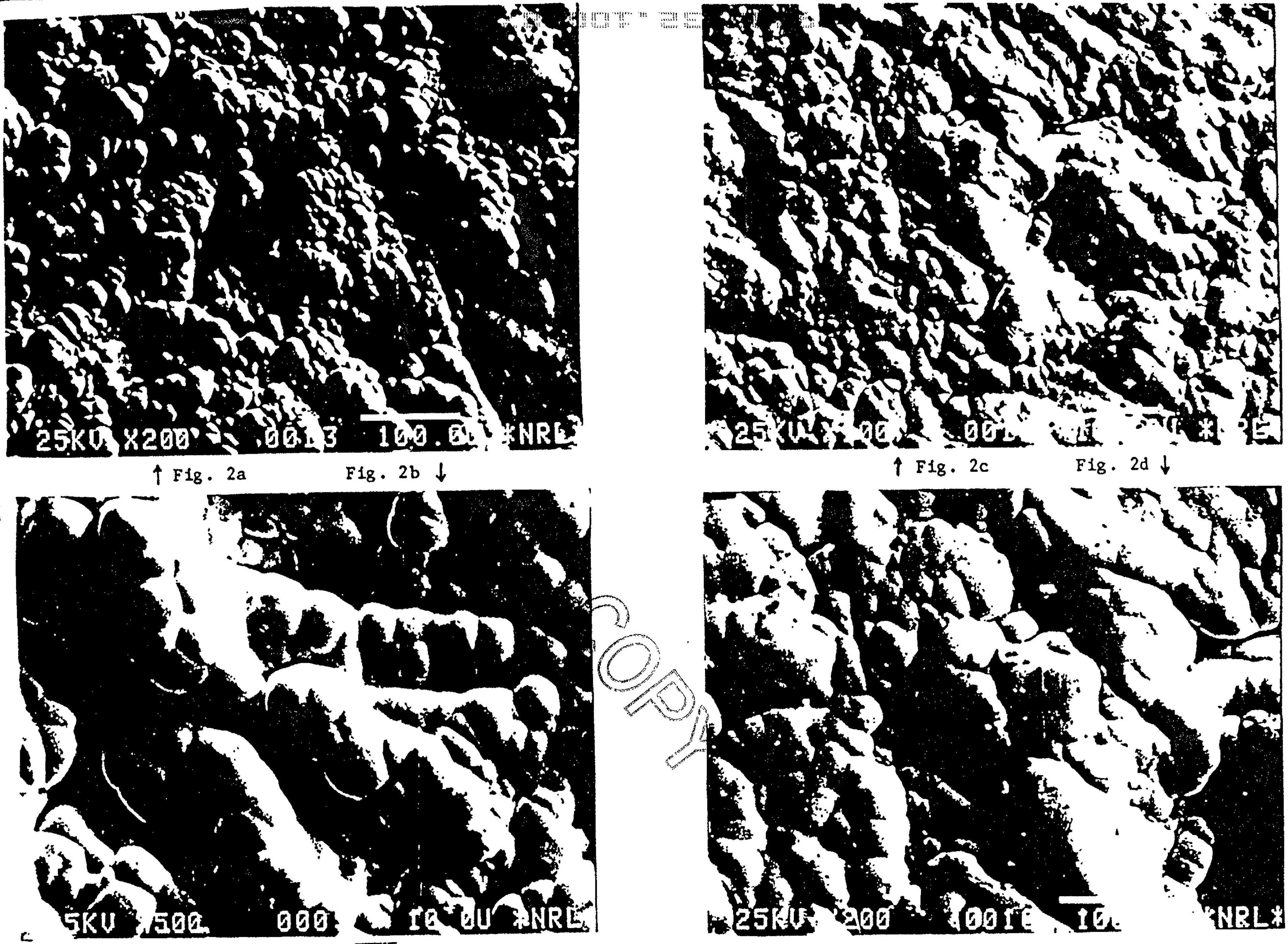

Figure 2: Scanning electron microscope pictures of the Labsphere 400 μin coating at 200X (Fig. 2a) and 500X (Fig. 2b). The newer coating used in the NRL sphere is shown at 100X magnification (Fig. 2c) and 200X magnification (Fig. 2d).

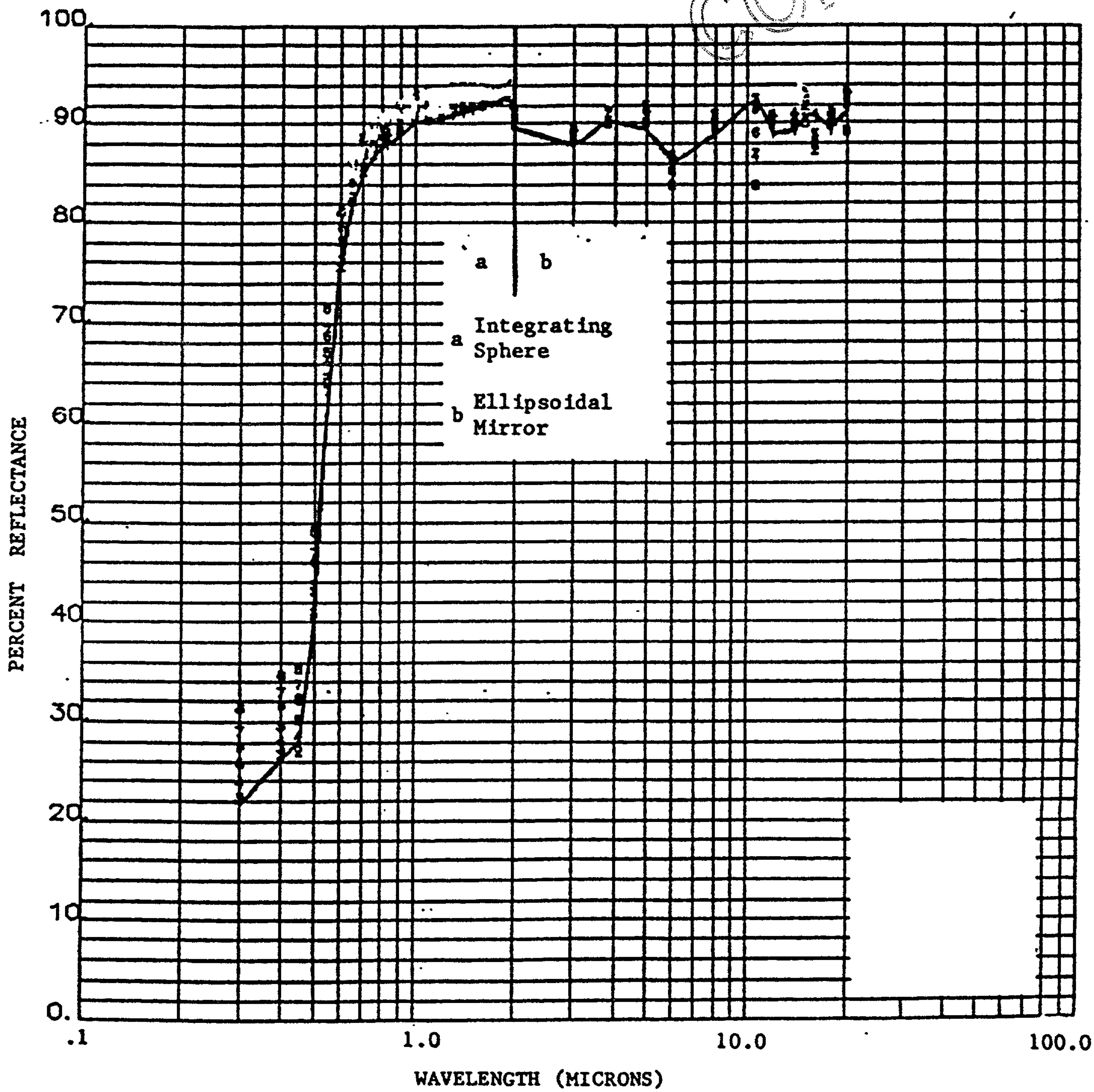

Figure 3: Total reflectance versus wavelength for Labsphere 400 micro-inch diffuse gold coating. The discontinuity at 2.0 microns marks the transition from an integrating sphere to an ellipsoidal mirror reflectometer.

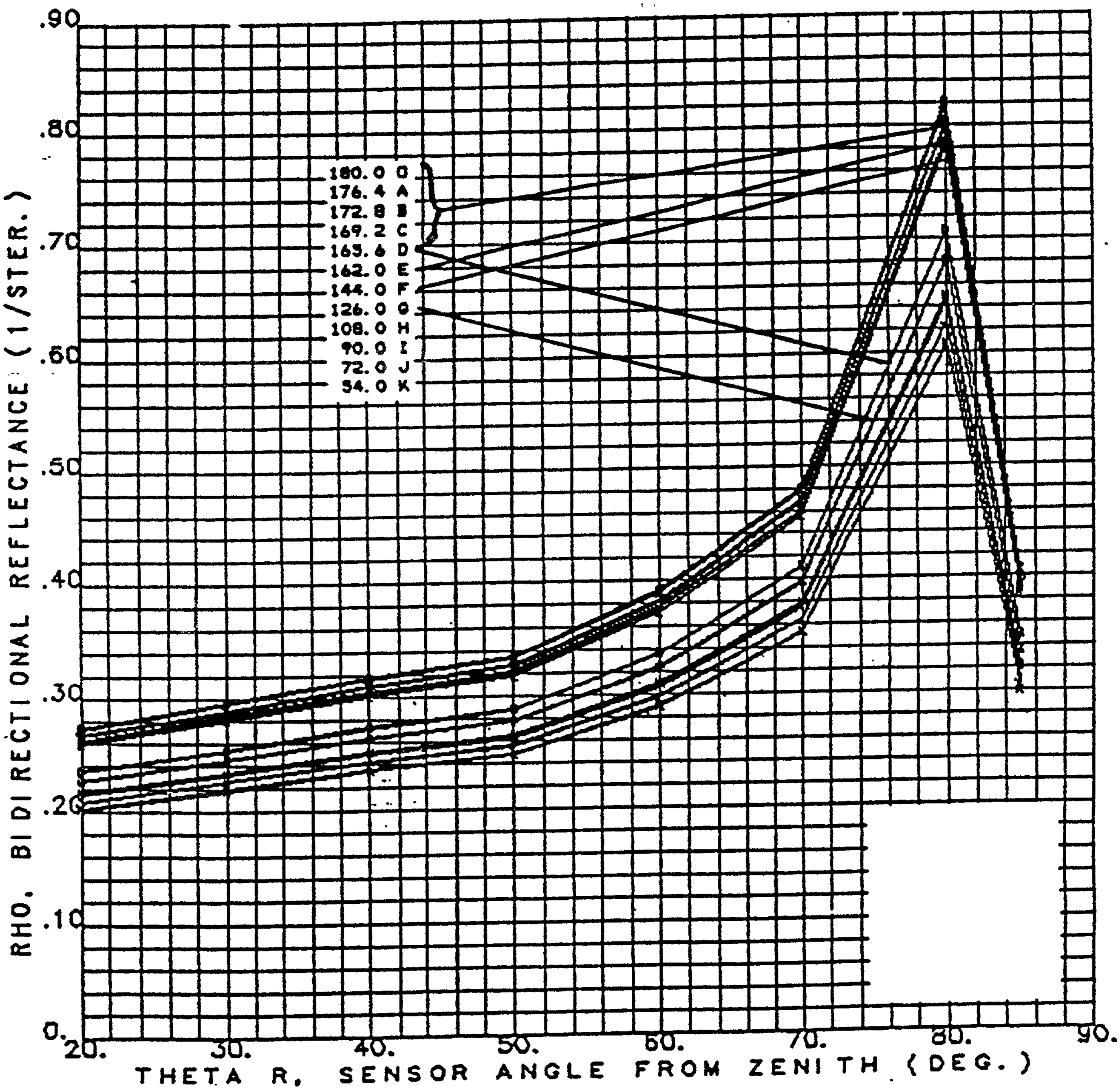

Figure 4: Bidirectional reflectance data for Labsphere 400 micro-inch diffuse gold coating. The data shown was measured at an incidence angle of 20 degrees, a wavelength of 10.6 microns, and a variety of angles in different azimuthal planes ranging from the plane of incidence (top curve) to 126 degrees out of the plane of incidence (bottom curve).

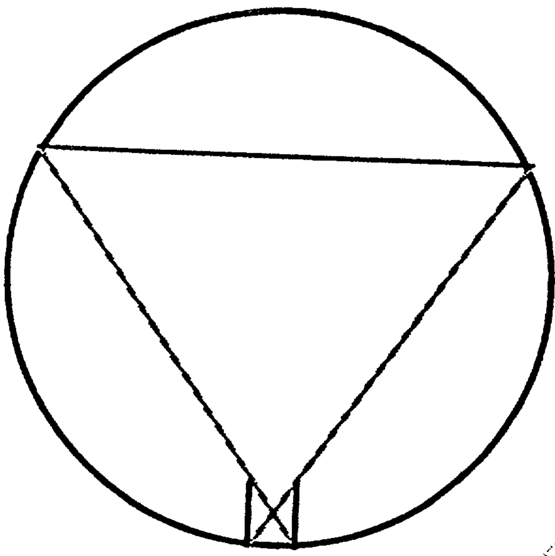

Fig. 5a

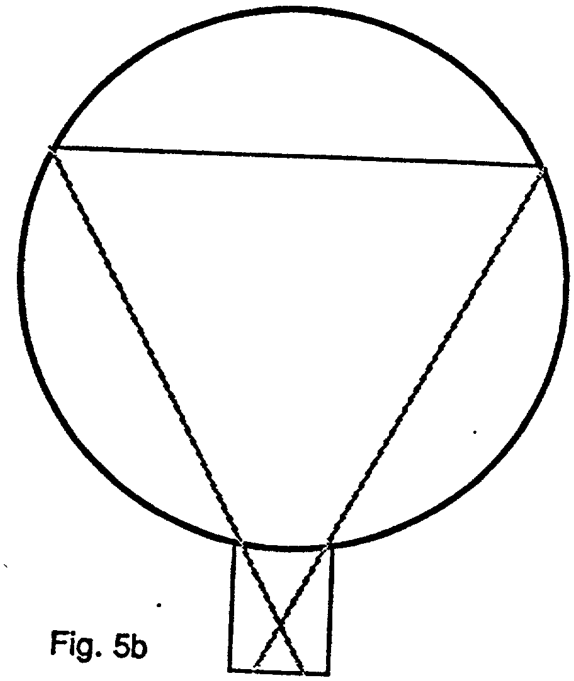

Fig. 5b

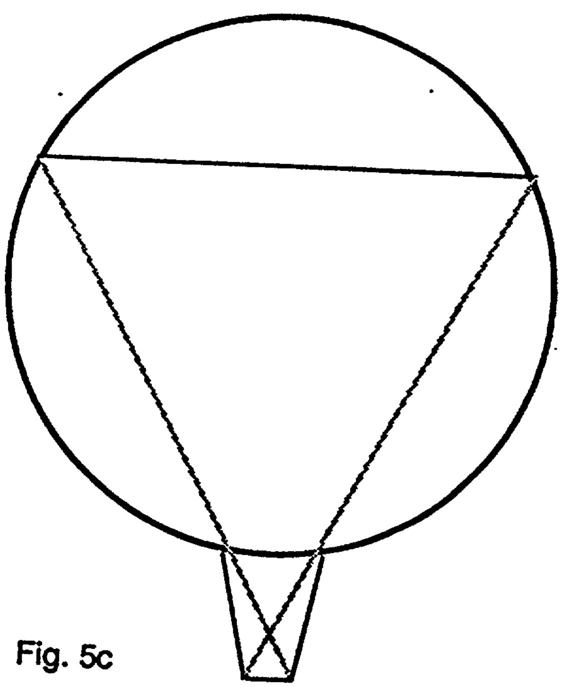

Fig. 5c

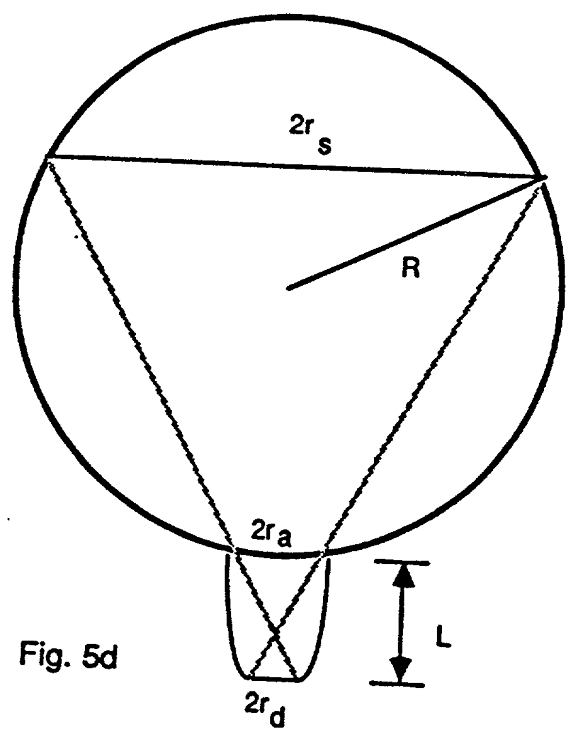

Fig. 5d

Figure 5: Detector optics coupling schemes: a) baffle, b) collimator, c) reflecting cone, and d) Compound Elliptic Concentrator (CEC).

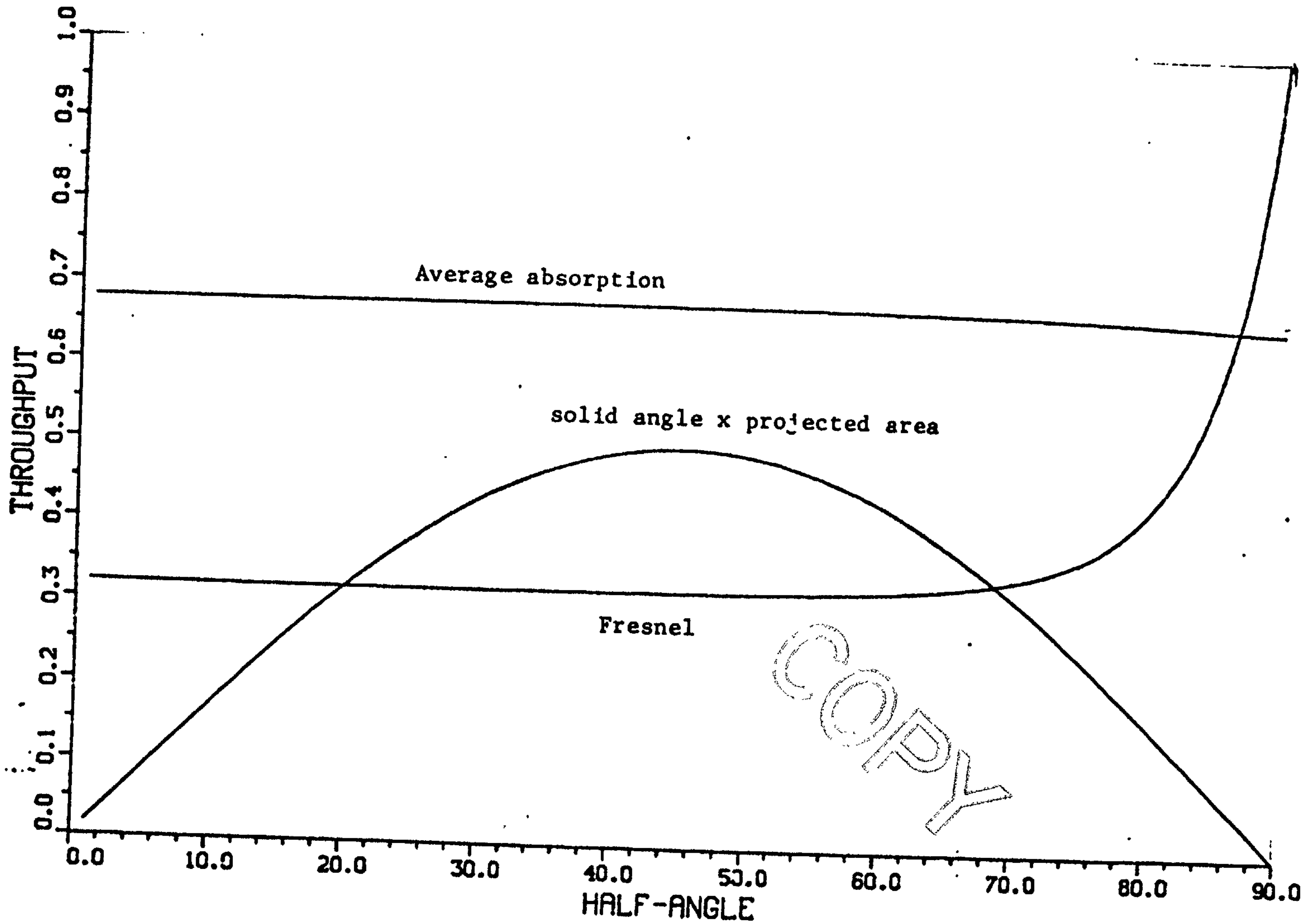

Figure 6: Average absorption vs. cone half angle for Lambertian illumination of a HgCdTe detector at 10 microns. The high Fresnel reflection losses at large angles do not decrease the average absorption significantly due to the projected area term in the weighting factor.

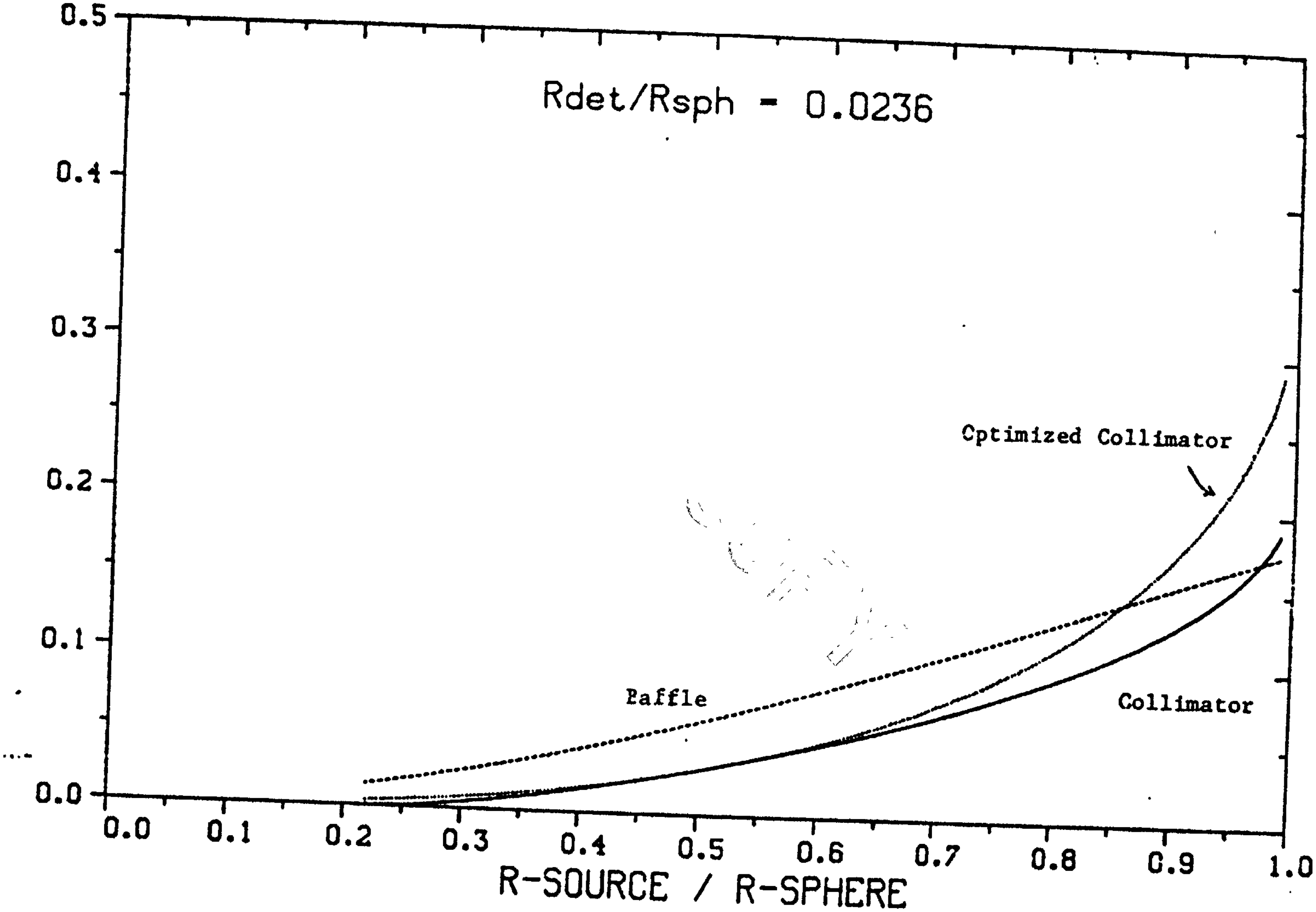

Figure 7: Integrating sphere detector optics throughput (normalized by CEC throughput) vs. source radius divided by sphere radius. For a source radius of 0.9, the CEC is over five times more efficient at collecting radiation than the optimized collimator.

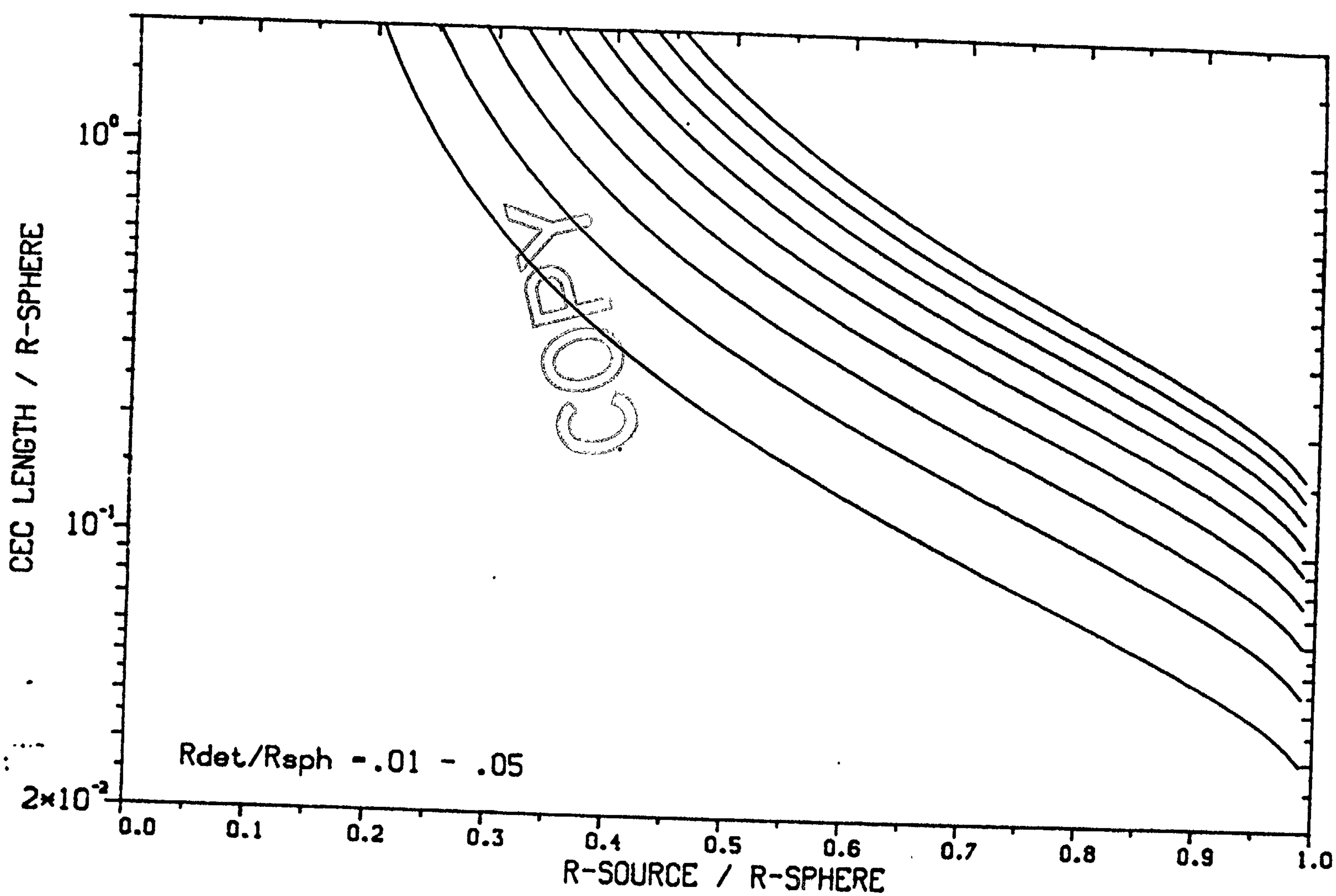

Figure 8a: CEC length (normalized by sphere radius) versus source size for detector radii ranging from .01 - .05 sphere radii. As the source size approaches the CEC aperture the length goes to infinity, as expected.

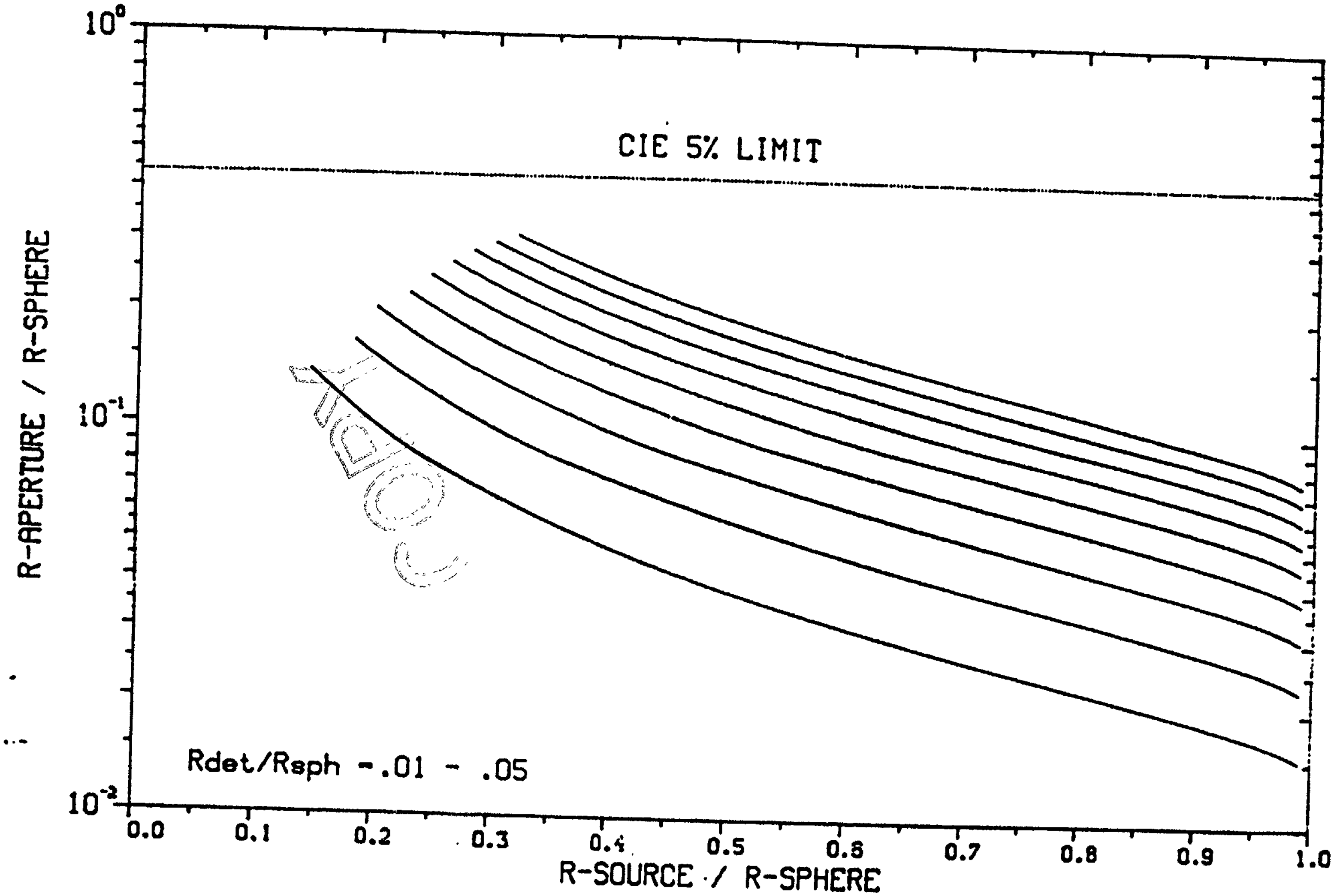

Figure 8b: CEC aperture radius (normalized by sphere radius) versus source size for detector radii ranging from .01 - .05 sphere radii. The curves stop where the aperture and source radii become equal; at that point the CEC length is infinity.

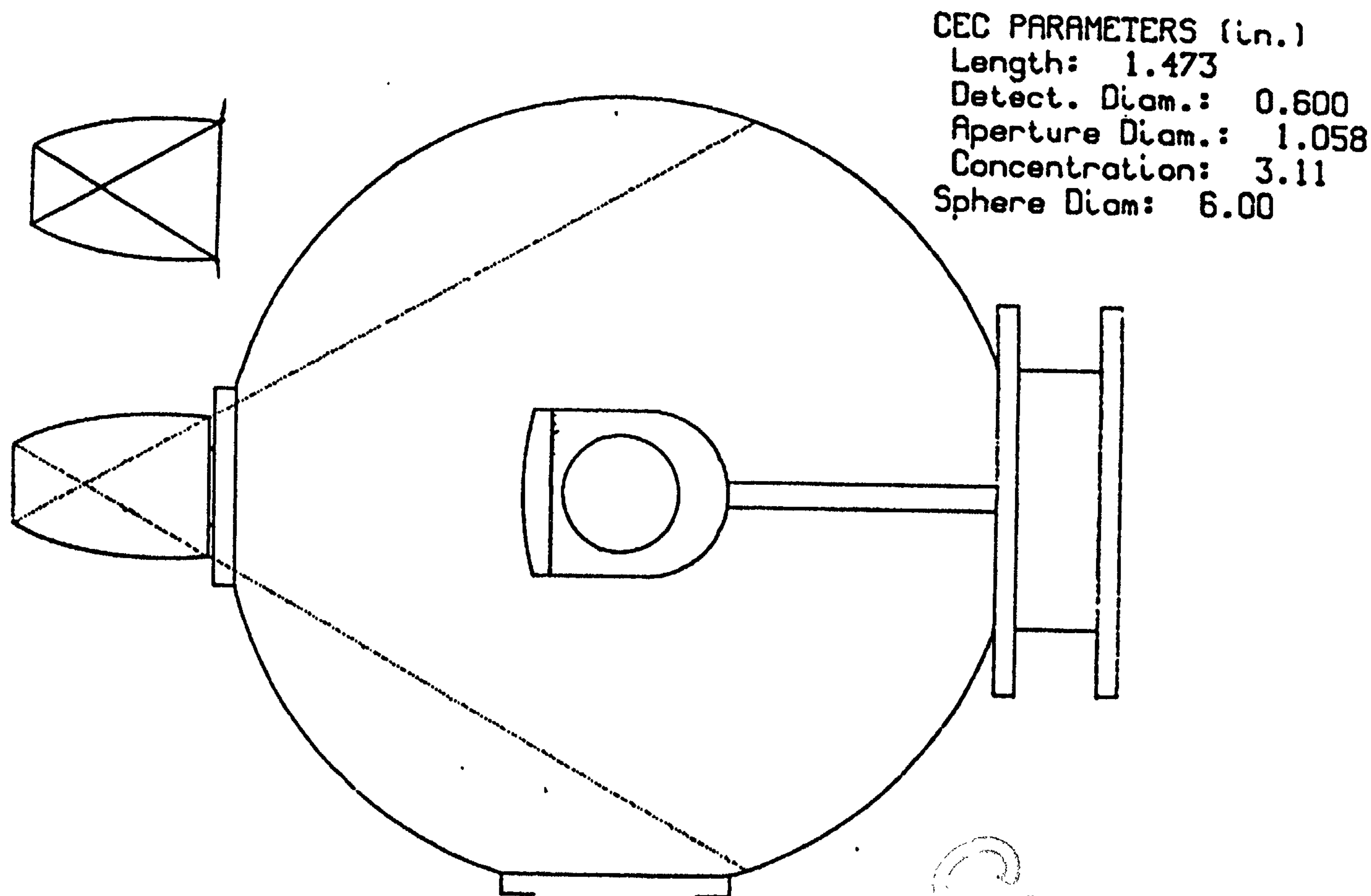

Figure 9: Effect of refraction compensation on the CEC length and concentration. The upper CEC was designed for the same source, sphere, and detector radii as the lower, however, no cover window over the detector was assumed. The concentration and length for the upper CEC are 2.98 and 1.399 respectively, or about 4-5% less than the refraction compensated CEC.

Obviously numerous additional modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that, within the scope of the appended claims, the invention may be practiced other than described specifically herein above.

10/9/87

ABSTRACT

Disclosed is an integrating sphere for measuring the diffuse reflectivity of material samples in the infrared. The sphere is disposed in an airtight vacuum chamber under an atmosphere that does not absorb in the infrared. The sphere has two positions at which a sample may be mounted, one on a rod at the sphere's center, another on the sphere's wall, each mounting position corresponding to two different modes of testing samples. The rod acts as a mounting pedestal for a center mounted sample, and is rotatably mounted about its elongate vertical axis so that the sample can rotate with the rod in a horizontal plane. The rod penetrates the sphere and the chamber, and terminates in a knob or handle by which the rod can be rotated to position the sample at a preselected angle. Adjacent to the position for wall mounting, there is a sample heater. This configuration allows one to measure diffuse reflectance of a sample as a function of incidence angle and temperature. In the center-mount configuration, the handle enables one to anglularly reposition a center mounted sample without the need to vent and replenish the chamber's atmosphere after each test.

Navy Case No. 70,840

WHAT IS CLAIMED AND DESIRED TO BE SECURED BY LETTERS PATENT OF THE UNITED STATES IS:

1. A system comprising:

an integrating sphere;

an airtight chamber means effective to provide the inside of said chamber means with a non-air atmosphere of preselected composition; and

a means for mounting a sample inside said integrating sphere.

2. The system of Claim 1, wherein said means for mounting is a pedestal effective to mount said sample at the center of said sphere, said pedestal means comprising an angular adjustment means for rotating said sample in said sphere, said angular adjustment means comprising a termination means for eambling said angular adjustment means to rotate said sample in said sphere from outside said chamber means.

3. The system of Claim 2, wherein said means set sample in subdivided into two sample portions set pedestal means comprises a mount effective to mount said sample portions back-to-back on said pedestal.

4. The system of Claim 1 wherein said means for mounting is a clamping means for mounting said sample on the inside wall of said integrating sphere.

5. The system of Claim 4 wherein said integrating sphere has a port passing through the wall of said sphere for mounting is effective to expose said sample to light flux incident upon said port from the inside of said sphere, and wherein said means for mounting comprises a clamping means for forcing said sample into fixed abutment with said port.

6. The system of Claim 5 wherein said means for mounting comprises a heating means for heating said sample, said clamping means is effective to mount said heating means in thermal contact with said sample effective to heat said sample to a preselected temperature, said clamping means being effective mount said heating means physically distant from said wall.

7. The system of Claim 6 wherein said clamping means is a tension spring having an arm effective to abuttingly clamp said heating means against said sample and said sample against said wall, at least a portion of said heating means being sandwitched between said sample and said arm.

8. The system of Claim 7 wherein said heater is an electrical resistive heater.

9. In an integrating sphere, said sphere having a wall and a port means for passing light flux from the inside of said sphere to the outside, a means for mounting a sample

effective to expose at least a portion of said sample to light flux exiting from said interior, said means for mounting comprising a clamping means for forcing said sample into fixed abutment with said port means.

10. The system of Claim 9 wherein said means for mounting is a pedestal effective to mount said sample at the center of said sphere, said pedestal means comprising an angular adjustment means for rotating said sample in said sphere, said angular adjustment means comprising a termination means for eambling said angular adujust means to rotate said sample in said sphere from outside said chamber means.

11. The system of Claim 10 wherein said set sample is subdivided into two sample portions set pedestal means comprises a mount effective to mount said sample portions back-to-back on said pedestal.

12. The system of Claim 11 wherein said means for mounting is a clamping means for mounting said sample on the inside wall of said integrating sphere.

13. The system of Claim 12 wherein said integrating sphere has a port passing through the wall of said sphere, said means for mounting is effective to expose said sample to light flux incident upon said port from the inside of said sphere, and wherein said means for mounting comprises a

clamping means for forcing said sample into fixed abutment with said port.

14. The system in Claim 13 wherein said means for mounting comprises a heating means for heating said sample, said clamping means is effective to mount said heating means in thermal contact with said sample effective to heat said sample to a preselected temperature, said clamping means being effective mount said heating means physically distant from said wall.

15. The system in Claim 14 wherein said clamping means is a tension spring having an arm effective to abuttingly clamp said heating means against said sample and said sample against said wall, at least a portion of said heating means being sandwiched between said sample and said arm.

16. The system in Claim 15 wherein said heater is an electrical resistive heater.

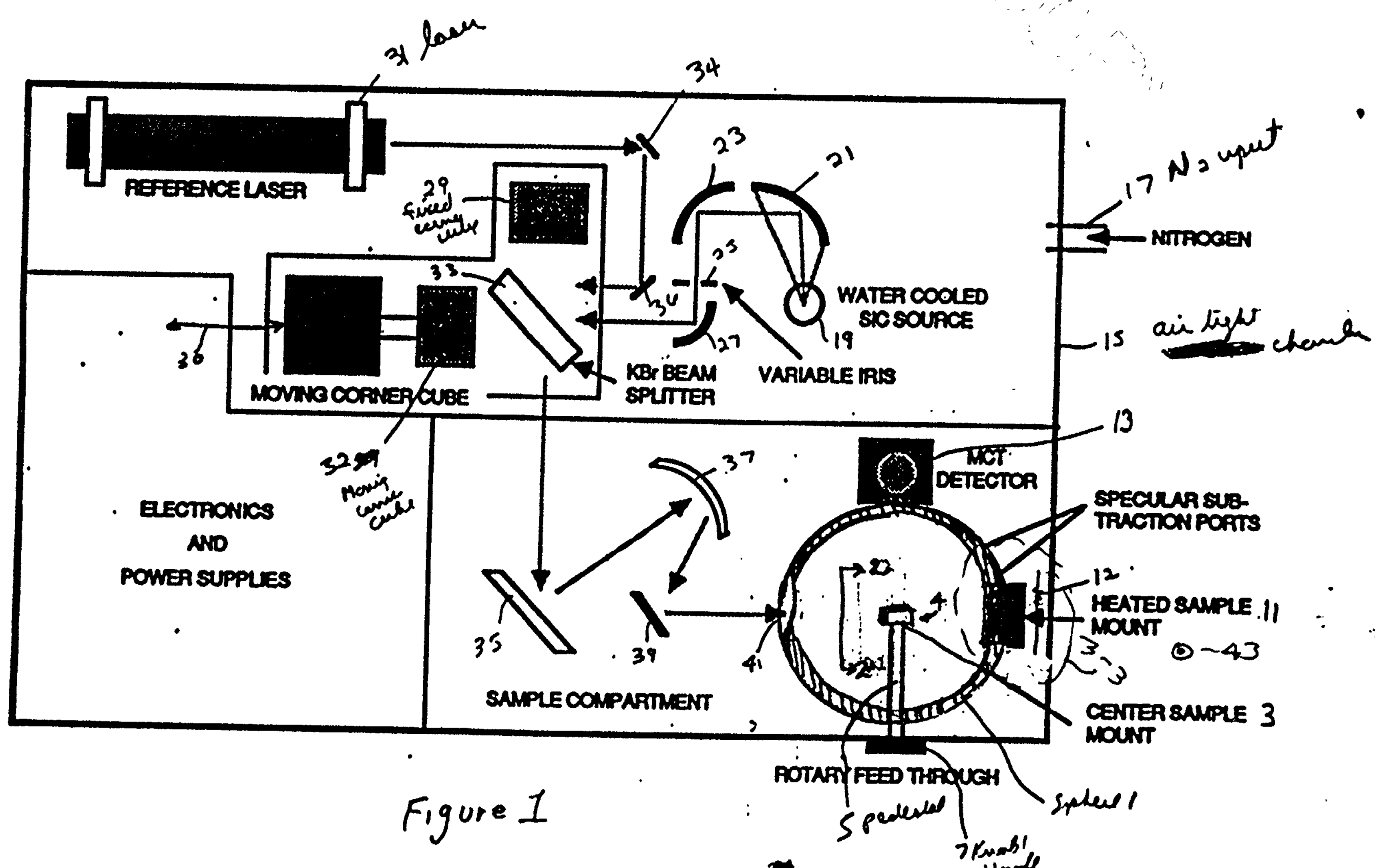
31 laser
REFERENCE LASER
34
23
21
17 N2 input
NITROGEN
29 fixed corner cube
33
25
34
WATER COOLED
SIC SOURCE
19
15
air tight chamber
36
MOVING CORNER CUBE
27
KBr BEAM
SPLITTER
VARIABLE IRIS
13
32 Moving corner cube
MCT
DETECTOR
37
SPECULAR SUB-
TRACTION PORTS
ELECTRONICS
AND
POWER SUPPLIES
12
HEATED SAMPLE 11
MOUNT
35
39
41
3-3
~43
SAMPLE COMPARTMENT
CENTER SAMPLE 3
MOUNT
ROTARY FEED THROUGH
5 pedestal
7 Knob/Handle
Sphere 1

Figure I

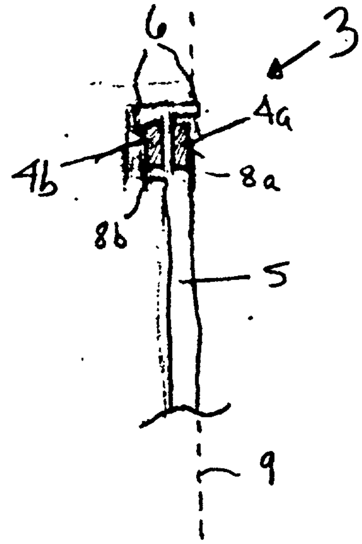

Figure 2.

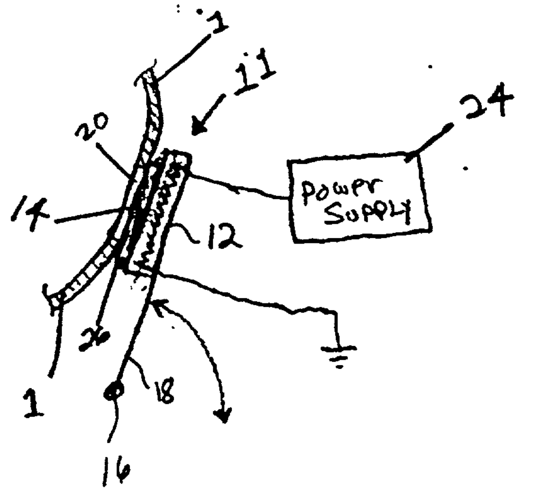

Figure 3

Navy Case 70,840

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application:
Keith A. Snail
Kevin L. Carr
For: INFRARED INTEGRATING SPHERE

Declaration of Keith A. Snail

Honorable Commissioner of Patents and Trademarks
Washington, D. C. 20230

Sir

I, the undersigned co-inventor with Kevin L. Carr of the above-styled application for Letters Patent, declare that:

1. I am, and was at the time of the making of the instant invention, an employee of the U.S. Navy; and the said Kevin L. Carr is, and was at the time of the making of the instant invention, an employee of Labsphere, Inc., of North Sutton, N.H.

2. I and the said Kevin L. Carr are the co-authors of the paper "Optical Design of an Integrating Sphere Fourier Transform Spectrophotometer (FTS) Emissometer" prepared for a conference sponsored by the SPIE, a professional organization, in May of 1986. A copy of said paper is appended hereto as "Appendex A".

3. At said conference, I presented a talk using a set of viewgraphs. At the conference I purposely refrained from distributing said viewgraphs or copies thereof, or

pre-prints or other copies of said paper, to anyone. A copy of said viewgraphs is appended hereto as Exhibit "B".

4. Said paper was first distributed by SPIE in volume 643 of the "SPIE Conference on Infrared Optics" on October 13, 1987, in support of which is appended hereto as Exhibit "C" a photostat of a letter I obtained from SPIE concerning the distribution date of volume 643.

5. I know of no distribution of either said paper or said viewgraphs before October 13, 1986, to anyone outside of the U.S. Navy or Labsphere, Inc.

I further declare that I am aware that willful false statements and the like are punishable by fine or imprisionment, or both (title 18 of the U.S. Code, section 1001), and may jeoparadize the validity of the application or any patent issuing thereon, that all statements in this declaration made of my own knowledge are true, and that all statements made on information and belief are believed to be true.

____________________ ____________________

Date | Keith A. Snail